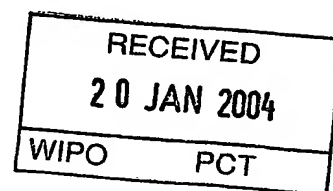




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I, JULIE BILLINGSLEY, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2003903167 for a patent by INTEC LTD as filed on 20 June 2003.



WITNESS my hand this
Fourteenth day of January 2004

J. Billingsley

JULIE BILLINGSLEY
TEAM LEADER EXAMINATION
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AUSTRALIA

Patents Act 1990

PROVISIONAL SPECIFICATION

Applicant:

INTEC LTD

Invention Title:

RECOVERY OF PRECIOUS METALS FROM SULFIDIC MATERIALS

The invention is described in the following statement:

RECOVERY OF PRECIOUS METALS FROM SULFIDIC MATERIALS

Field of the Invention

The present invention relates to a process for recovering precious metals, such as gold, from a sulfidic material. The invention can be applied to both un-contaminated and contaminated sulfidic materials, including those having a relatively high carbon content (a so-called "double-refractory material") or no or low carbon content (a so-called "single-refractory material"). When the term "relatively high carbon content" is used herein it refers to a carbon content in the sulfidic material that is typically higher than about 2 wt%.

Background to the Invention

Across the world there are significant deposits and quantities of sulfidic materials including economically desirable metals to recover, especially precious metals such as gold and silver. For example, there are significant deposits and stockpiles of pyritic ores including gold and/or silver and other precious metals such as platinum and platinum group metals.

Some of these deposits are contaminated with difficult to process contaminants such as arsenic, antimony, bismuth or other heavy metals. Ore treatment may also be complicated when high levels of carbon are present, as carbon associates with and has a high affinity for precious metals such as gold.

Current commercially available options for the oxidation of sulfidic materials include roasting, pressure oxidation (POx) and bio-oxidation (Biox). In the POx and Biox processes a sulfate medium is typically employed.

Roasting sulfidic ores presents significant problems due to emissions of environmentally toxic sulfur based gases (so-called SOx gases). Where arsenic is present in the ore, poisonous substances such as arsenic trioxide are produced. For these reasons international trends are to move away from roasting of sulfide ores.

Pressure oxidation of sulfidic materials is employed to avoid the problems of roasting, but requires high pressures (typically greater than 30 bar) and relatively high temperatures (greater than 200°C). Pressure oxidation is also typically carried out in a sulfate based solution.

US6461577 discloses a bio-oxidation process for treating sulfidic materials containing arsenic where the sulfidic material is subjected to a two-stage Biox process to solubilise the arsenic. The configuration of the leaching process is complex, as is the use of bio-leaching bacteria. In addition, bio-oxidation is notoriously slow.

US4053305 discloses a leaching process for the recovery of copper and silver

from a sulfide ore using a combination of ferrous chloride solution and pressurised oxygen. Whilst copper is dissolved in the leach, silver is deliberately not leached and is passed with the solid residue from the leach. The silver must then be extracted from the residue using sodium cyanide, an environmentally harmful leaching agent.

5 US4410496 discloses a leaching process for the recovery of copper, lead and zinc from a sulfide ore using a combination of calcium or barium chloride solution and pressurised oxygen. Again, precious metals in the ore remain unleached and pass with the solid residue from the leach and must be separately extracted.

10 US4655829 discloses a leaching process for the recovery of metals from a sulfide ore that comprises arsenic and antimony. In this process a bulk sulfide concentrate is prepared from the arsenic sulfide ore. The concentrate is slurried in excess calcium chloride solution. Once the concentrate is prepared, the total metal content and composition of the concentrate needs to be determined. To prevent soluble arsenic compounds or toxic arsenic vapours being created in the process the concentrate
15 is blended with a balancing solution slurry containing a predetermined concentration of copper, lead, zinc, or a mixture thereof in the form of sulfides of those metals. In this regard, the concentrate and the balancing solution slurry are combined to form a reaction slurry having a predetermined metal content such that the molar concentration of arsenic and antimony in the mixture is about equal to the molar concentration of
20 copper, lead, and zinc, ranging from between about 60-40 or 40-60. Only once the mixture is properly balanced is it heated and aerated under pressure to oxidise the metals to soluble components. In other words, the balancing is essential so that no soluble arsenic compounds or toxic arsenic vapours are created.

25 It would be advantageous if a simple hydrometallurgical process could be provided for recovering a precious metal, especially gold, from a sulfidic material.

Summary of the Invention

In a first aspect the present invention provides a process for recovering a precious metal from a sulfidic material comprising the steps of:

- 30
- preparing an acidic aqueous halide solution having an oxidation potential sufficient to oxidise the sulfidic material and render the precious metal soluble in the solution;
 - adding the material to the acidic aqueous halide solution so that the sulfidic material is oxidised and the precious metal is solubilised; and
 - separating the precious metal from the oxidised sulfidic material.

35 The present inventors have surprisingly discovered that when a sufficient oxidation potential is maintained in the acidic halide solution, the sulfidic material can be oxidised simultaneously with precious metal solubilisation in a single stage.

Furthermore, the inventors have surprisingly discovered that when the sulfidic material is contaminated with arsenic, antimony or the like the precious metal can be solubilised whilst the arsenic etc can be simultaneously leached and precipitated in a single stage without the need for a prior or initial solution balancing step.

5 In this regard, in a second aspect, the present invention provides a process for recovering a precious metal from a sulfidic material contaminated with arsenic etc comprising the steps of:

- preparing an acidic aqueous halide solution having an oxidation potential sufficient to oxidise the sulfidic material and render the precious metal soluble in the solution, and
- 10 having a pH at which the arsenic is precipitated;
- adding the material to the acidic aqueous halide solution so that the sulfidic material is oxidised, the precious metal is solubilised and the arsenic is precipitated; and
- separating the precious metal from the oxidised sulfidic material and precipitated arsenic.

15 The process of the first and second aspects also differs from the POx and Biox processes in that a halide rather than sulfate-based leaching solution is employed.

The inventors have noted that halides (like cyanide) form strong complexes with precious metals such as gold and can thereby facilitate precious metal dissolution and subsequent precious metal recovery by eg. carbon adsorption. However, because

20 halides are weaker ligands than cyanide, the inventors have developed a processology in which a sufficiently high oxidation potential (Eh) in an acidic environment (preferably of pH <3) achieves a dissolution capability of the precious metal similar to cyanide.

Advantageously, the process can be operated in a closed loop or recycle mode with attendant economic benefits (eg. simplicity, low energy consumption, preservation

25 of mass balance etc.). The inventors have also observed that the process can be applied to recover precious metals from any sulfidic material, including otherwise difficult to treat ores and concentrates such as double-refractory materials having a relatively high carbon content (eg. carbon-containing arsenopyrites).

Preferably the solution bearing the precious metal is separated from the

30 oxidised sulfidic material and precipitated arsenic (when present) in a solid-liquid separation stage, and the precious metal is then recovered from the solution in a metal recovery stage, preferably by adsorption onto activated carbon, typically in one or more carbon-containing columns. Preferably after precious metal adsorption onto activated carbon the carbon is removed and burnt to recover the precious metal, or is eluted with

35 a cyanide solution and the eluate passed to an electrolysis stage for recovery of the precious metal by electro-winning. In this regard, the present process advantageously differs from current commercial processes, where cyanidation of the oxidation residue

is required for precious metal (gold) extraction, requiring a separate dedicated leach circuit. In the present invention the precious metal is already solubilised in the leach, so cyanide leaching is not required. In addition, many environmental authorities now require the destruction of residual cyanide, especially in environmentally sensitive locations, which can add additional costs.

In the case of an un-contaminated sulfidic material (eg. a single refractory pyritic ore uncontaminated by arsenic etc) the oxidation of the sulfidic material is typically performed in one stage. In the case of a contaminated sulfidic material (eg. a single or double refractory pyritic ore contaminated by arsenic and/or carbon etc) the oxidation of the sulfidic material is typically performed in two stages, although precious metal solubilisation is achieved in a first of these stages.

Typically the solution is recycled to the sulfidic material oxidation stage after it has passed through the metal recovery stage. Preferably the metal recovery stage is provided in-line, after the solid-liquid separation stage, and prior to solution recycle to the sulfidic material oxidation stage. Use of the terminology "in-line" refers to a stage that is provided as part of a solution circuit (ie. the "circuit" resulting from solution recycle to the sulfidic material oxidation stage). In addition, metal recovery processes other than carbon adsorption may be employed including ion exchange, solvent extraction, etc.

In the case of a double-refractory ore that includes carbon, an additional separate metal recovery stage may need to be provided (ie. separate to the solution recycle circuit) to recover any precious metal that passes with material solids from the sulfidic material oxidation stage. This separate stage may be required because some of the precious metal (eg. gold) passes with the carbon right through the oxidation process and is not solubilised. The separate metal recovery stage may employ a conventional roasting or smelting process and optionally leaching (eg. using solution from the sulfidic material oxidation stage) may be employed after roasting to recover any remaining precious metal in the roasted solids material (eg. gold).

Typically the precious metal to be recovered is gold, but can be silver, platinum or another platinum group metal, the recovery of which metal typically justifies the process economics.

Most preferably the aqueous halide solution is a soluble metal halide solution, typically having a halide concentration of approximately 8 moles per litre. Preferably the halide is chloride, but can be bromide or a mixture of halides such as chloride and bromide.

The process is preferably operated such that the metal in the dissolved metal halide solution functions as a multi-valent species. In this regard, a multi-valent species

is typically selected that has both a relatively high oxidation state to participate in oxidation of the sulfidic material and a relatively lower oxidation state to which it is reduced during oxidation. Advantageously, the multi-valent species can then be regenerated to its relatively high oxidation state, whereafter the regenerated multi-valent species can be recycled to the sulfidic material oxidation stage to participate in further oxidation. Advantageously, the regeneration of the multi-valent species occurs during the leaching stage(s) so that the regenerated species can be recycled to the sulfidic material oxidation stage as part of the preferred closed loop or recycle mode of the process, with the attendant economic benefits (eg. preservation of mass balance, simplicity, low energy consumption, etc.).

Typically the metal in the metal halide solution is copper, but may also be iron etc. Either of these multi-valent species effectively acts as an electron transfer agent. For example, in the solution recycled to the sulfidic material oxidation step the metal is in its relatively high oxidation state (eg. Cu(II) or Fe(III)), and after oxidation is in its relatively lower oxidation state (eg. Cu(I) or Fe(II)). In the leaching stages the multi-valent species typically exists as a couple (ie. in its high and low oxidation states). However, other multi-valent species may be employed including possibly cobalt, manganese and vanadium.

Where the sulfidic material is an arsenopyrite, by controlling the oxidation potential, arsenic can be leached into solution in a first leaching stage. However, preferably the solution pH is controlled such that, once leached, the arsenic precipitates as ferric arsenate (scorodite). Again, where the sulfidic material is an arsenopyrite, preferably the pyrite component is leached in a second leaching stage in which the solution pH is also controlled to maintain arsenic as a ferric arsenate precipitate. Thus, the arsenic passes out of the process with the solid residue at the solid-liquid separation stage and does not interfere with precious metal recovery.

For an un-contaminated single-refractory pyritic material the sulfidic material oxidation step typically comprises a single leaching stage in which the pyritic material is oxidised and the precious metal simultaneously solubilised.

Each leaching stage may be operated co- or counter-currently and in this regard, each stage may comprise one or more vessels.

Preferably the entire solution from the first leaching stage is fed to the second leaching stage.

When the sulfidic material is contaminated with eg. arsenic, typically in the first leaching stage the material is contacted with solution at an Eh sufficient to leach the contaminant and solubilise the precious metal (eg. gold), preferably at an Eh of around 0.7-0.8 volts (ref. SHE). At this solution Eh the pyrite component of the material

is not substantially leached. Preferably in the first leaching stage the solution pH is less than 1 but greater than about 0.5 so as to precipitate the contaminant immediately after it is leached. Preferably in the first leaching stage the solution temperature is about 80-105°C, more typically 80-95°C.

For an un-contaminated sulfidic material (where a single leaching stage is employed) or for the second leaching stage employed for leaching the pyrite component of a contaminated sulfidic material, typically the material is contacted with a solution having an Eh sufficient to leach pyrite, preferably an Eh of around 0.8 - 0.9 volts. Again, typically the solution pH is less than 1 but is greater than about 0.2 so as to precipitate the contaminant immediately after it is leached. Also, for pyrite leaching, typically the solution temperature is the same or higher than for arsenopyrite leaching, typically about 90°C to 105°C.

To achieve a higher solution Eh in the single or second leaching stage, it may be necessary to add additional oxidant such as oxygen, air, chlorine gas, hydrogen peroxide etc. To achieve optimal solution pH to maintain the contaminant in its precipitated form and to regenerate cupric ion an acid such as sulfuric acid and/or a base such as calcium carbonate may need to be added to the single or second leaching stage to lift the pH, otherwise arsenic and iron will not precipitate and rather will solubilise. In this regard, in either the single or second leaching stage, oxidation of the pyritic component of the material may produce sufficient or excess sulfuric acid. Alternatively, hydrochloric acid or any other acid which does not interfere with the process chemistry can be employed.

Whereas the separated solution after leaching is passed to precious metal recovery, the separated residual solids are typically passed to disposal.

Preferably after precious metal recovery a solution conditioning stage is employed to remove (precipitate) ferric sulfate and thus control the level of this species in the process. Typically in this stage limestone and calcium carbonate are added to the solution to form a hematite/gypsum precipitate which is then filtered and disposed of with the solids residue from the leaching stage(s). However, ferric removal is preferably controlled by regulating limestone addition to maintain some iron in solution, which in turn prevents cupric copper precipitation (ie. because iron precipitates at a lower pH than copper and buffers the pH whilst it precipitates, thereby acting as a safeguard against copper precipitation).

Preferably in the solid-liquid separation stage solids residue is filtered from the solution, however other separation methodologies may be employed such as solid/liquid settling, solution evaporation, centrifugation etc.

When a high level of carbon is present in the sulfidic material (eg. 2-20 wt% carbon), a surfactant such as a blinding agent can advantageously be added to the solution during sulfidic material oxidation to prevent precious metals (such as gold) from adsorbing onto carbon in the material. The blinding agent is typically one or more organic solvents including kerosene, phenol ethers, etc. Alternatively, activated carbon can be added to the solution to preferentially adsorb gold. The use of a blinding agent or activated carbon may obviate the need for a separate metal recovery stage to separate any precious metal which may otherwise pass with the carbon in the solids residue.

A most advantageous application of the present process is in relation to the recovery of precious metals from pyritic ores and concentrates, where typically the contaminant is arsenic, antimony, bismuth, mercury, cadmium, etc and which occur naturally in many as-mined pyritic materials.

Other economically significant metals may additionally be recovered in the process including copper, nickel, zinc, lead etc. In addition, in certain applications, the contaminant may itself be desirable or necessary to recover. For example, the contaminant may be economically valuable or environmentally harmful, prompting its recovery from the contaminant precipitate (eg. this may be the case for a contaminant such as antimony, bismuth, cadmium etc.).

Brief Description of the Drawings

Notwithstanding any other forms which may fall within the scope of the present invention, preferred forms of the invention will now be described, by way of example only, and with reference to the accompanying drawings in which:

Figure 1 schematically depicts generalised process representations of the prior art POx and Biox processes, in comparison to a preferred process (IRGP) according to the present invention for recovering a precious metal from a sulfidic material;

Figure 2 schematically depicts a generalised process flow diagram for the recovery of a precious metal (gold) from a contaminated sulfidic material (arsenopyrite - FeAsS); and

Figures 3 & 4 plot gold and iron extraction, and solution Eh against time for various stages of the IRGP.

Modes for Carrying out the Invention

Prior to describing preferred processes of the invention by way of detail and example, preferred processes according to the present invention will first be described in overview and in context with the prior art POx and Biox processes by reference to Figure 1.

Preferred processes according to the present invention are referred to in this detailed description as the Intec Refractory Gold Process (IRGP). These processes were developed as a halide based alternative for the recovery of gold from refractory sulfide mineral deposits. A known treatment of such deposits is generally by way of flotation of ground ore to produce a concentrate, which is subsequently treated to oxidise sulfide minerals predominantly to sulfate, culminating in the extraction of the gold from the oxidation residue using cyanide.

Current commercially available options for the oxidation of sulfide minerals include roasting, pressure oxidation (POx) and bio-oxidation (Biox). The context of the IRGP by comparison to current hydrometallurgical practice (POx and Biox) is schematically depicted in Figure 1. The IRGP advantageously differs from the hydrometallurgical POx and Biox options in that a halide rather than sulfate medium is used. Gold is insoluble in sulfate, whereas halides, like cyanide, form strong complexes with gold to facilitate its dissolution and subsequent recovery by adsorption onto activated carbon. As halides are weaker ligands than cyanide an acidic environment (pH < 2) and higher solution temperature and potential (Eh) is employed to achieve the same gold extraction efficiencies.

When treating refractory sulfides, the use of a halide medium at certain solution oxidation potentials allowed sulfide oxidation to be performed concurrently with gold dissolution. After the gold laden solution was separated from the oxidized sulfide mineral slurry, the dissolved gold was able to be recovered by adsorption onto activated carbon, which was subsequently burnt, or eluted with cyanide for the ultimate recovery of gold metal by electrowinning. Unlike current commercial practice, the IRGP did not require cyanidation of the oxidation residue for gold extraction, which requires a separate dedicated leach circuit and possibly also the costly requirement for residual cyanide destruction.

There are a number of factors that can render a gold-bearing ore refractory, as shown in the following table:

Type	Causes of Refractory Characteristics
Liberation	Physical locking in silicates, sulphides, carbon, etc.
Occlusion	Passivation due to formation of a chemical layer.
Chemistry	Formation of auriferous compounds e.g. gold tellurides and aurostibnite.
Substitution	Elemental replacement by gold in mineral lattice e.g. "solid solution" gold in pyritic ores.
Adsorption	Adsorption of dissolved gold by 'active' carbonaceous material in the ore pulp.

The IRGP was developed specifically to treat concentrates produced from those refractory ores falling into the latter two categories of "substitution" and "adsorption". The major proportion of the world's gold reserves fall into these two categories, which are dominated by iron sulfides such as arsenopyrite and pyrite, occurring either separately or more commonly in combination. The IRGP was also applicable when "active" carbon was also present in the ore.

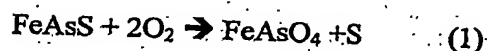
The IRGP process and chemistry are now described for the treatment of refractory gold concentrates containing the following mineral types:

1. Arsenopyrite
2. Arsenopyrite plus pyrite
3. Arsenopyrite plus pyrite plus carbon.

Arsenopyrite Oxidation Chemistry

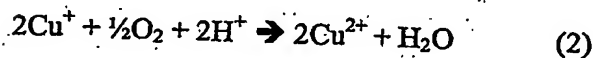
The presence of arsenic in refractory gold concentrates is chiefly in the form of arsenopyrite (FeAsS). Gold is typically "locked" in this arsenopyrite principally as a lattice-bound species, often referred to as a solid solution, rather than as native gold. Consequently gold liberation required complete destruction of the arsenopyrite lattice.

Destruction of the arsenopyrite lattice in the IRGP was achieved by chemical oxidation according to the following overall reaction:

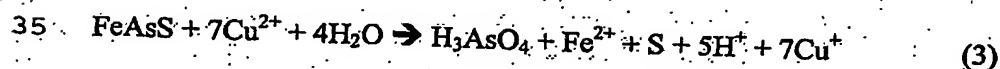


The oxygen did not oxidize the arsenopyrite directly, but acted through several intermediate steps, as its solubility in the process liquor was exceedingly low.

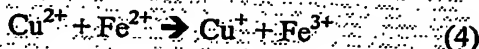
The oxygen was supplied directly from air sparged into the leach at atmospheric pressure, and initially was used to generate a soluble oxidant in the form of cupric ion (Cu^{2+}) according to the following reaction:



This reaction took place at the interface between the air bubbles and the process liquor. The cupric ion then oxidized the arsenopyrite according to the following reaction:



The ferrous and cuprous reaction products were subsequently oxidized by further air sparging according to reaction (2) and the following reaction:



In the presence of ferric ion, the arsenic acid readily formed insoluble ferric arsenate according to the following reaction:



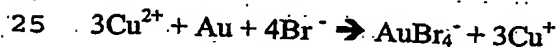
Ferric arsenate formed in the high chloride electrolyte and under the operating conditions used in the IRGP was typically crystalline and stable in the environment.

The action of the $\text{Cu}^{2+}/\text{Cu}^+$ couple was supplemented by the $\text{Fe}^{3+}/\text{Fe}^{2+}$ couple as a small background concentration of iron was always present in the process liquor.

The potential achievable under the influence of the Cu^{2+} and Fe^{3+} was in the region of 850mV (versus SHE) in the presence of oxygen. This potential was sufficient for the dissolution of gold, due to the stabilisation of the gold by the formation of a chloride complex according to the following reaction:



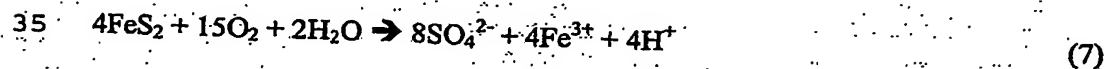
Where bromide was present in the process liquor, a gold-bromide complex was also formed according to the following reaction:



The oxidation was carried out at a temperature of 90-95°C in an 8M chloride electrolyte containing 20-40g/l Cu^{2+} ion plus 2-5g/l Fe^{3+} ion.

30 Pyrite Oxidation Chemistry

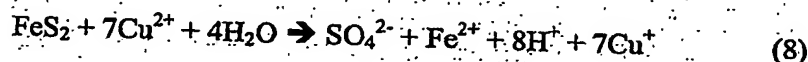
The oxidation of pyrite (FeS_2) in the IRGP was achieved via the same series of intermediate reactions as employed for arsenopyrite oxidation according to the following overall reaction:



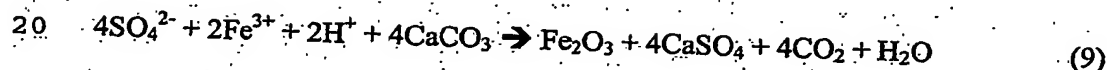
It was noted that the pyritic sulfur was oxidized all the way to sulfate in contrast to the arsenopyritic sulfur that was only oxidized to the elemental state.

Pyrite is more refractory than arsenopyrite, requiring a finer grind size to achieve acceptable reaction kinetics as explained below. However, individual pyrite samples exhibited variable reactivity that was thought to be influenced by arsenic substitution for a portion of the sulfur in the crystal lattice. Such pyrite is often termed arsenical pyrite and, the higher the arsenic contamination, the more the pyrite reactivity approached that of true arsenopyrite with an As/S ratio of one.

The reaction proceeded through the $\text{Cu}^{2+}/\text{Cu}^+$ couple as for arsenopyrite at a temperature of 90-95°C in the same liquor used for arsenopyrite oxidation according to the following reaction:



The Cu^+ and Fe^{2+} were oxidized by further oxygen sparging according to reactions (2) and (4). The ferric sulfate formed was precipitated as hematite and gypsum by the addition of limestone at a pH of approximately 1-1.5 according to the following reaction:



Limestone addition was controlled to maintain soluble iron in the range 2-5 g/l, which prevented the precipitation and loss to the leach residue of cupric copper.

25 Concentrate Grind Size

Concentrates for use in the IRGP were typically received in the size range of 80% less than 70-100 microns. Tests indicated that reaction kinetics were significantly enhanced when the concentrates were reground to a finer size (dependent on the characteristics of each individual concentrate). Where arsenopyrite was the sole gold-bearing mineral, a size of 80% less than 30-40 microns proved adequate to achieve good gold extraction and an acceptable leach retention time.

Where gold was locked in pyrite, the grind size principally depended on the reactivity of the pyrite which, as previously explained, varied greatly. For a highly active pyrite, the grind employed for arsenopyrite was used, but more refractory examples required finer grinding. This sometimes extend to an ultra-fine grind with 80% less than 6-10 microns in a more extreme refractory case. The inventors noted that

ultra-fine grinding technology has developed over the last 10 years to the point where many such mills are successfully operating at mines around the world.

Gold Recovery

The gold-bearing leach solution was passed through columns containing activated carbon onto which the gold was adsorbed. Retention time for gold adsorption was 10-15 minutes, which was similar to conventional practice for cyanide systems. Gold loading onto the carbon was typically 2-5% w/w due to the relatively high gold concentrations in the solutions (typically 10-100mg/l), as a consequence of the typical high gold grade of the concentrate. Gold recovery at such loading was via destruction of the carbon by combustion in a kiln. At lower loading, elution with cyanide followed by reactivation of carbon was more economic.

Impurity Management

The presence of impurities in the feed concentrate (such as Cd, Mn, Mg, etc.) had no detrimental effect on either the leaching or precipitation operations. Nevertheless, a method for the management of impurities was employed. This was achieved via precipitation from a bleed of the regenerated cupric solution with the purified brine returned to the process. Importantly the IRGP did not generate any liquid effluents and that all impurities are produced as solid by-products.

Limestone is added to the bleed to adjust the pH to 3.5, precipitating residual iron and copper, which are removed by filtration and recycled to the leach. Impurities, such as Cd, Mn and Mg, are then removed via slaked lime addition at pH 9 to form insoluble oxides that are recovered by filtration for disposal.

In the context of process equipment the IRGP was similar to Biox processes in that atmospheric pressure was used, but retention time was advantageously lower, typically in the range 6-20 hours. With pyrite oxidation a leach temperature higher than Biox was employed, but avoidance of an oxygen plant (as used for Pox) was achieved where the concentrate fed to the process was finely ground, typically to an ultra-fine level of eg. less than 10 μm . Materials of construction of process apparatus were fibre-reinforced plastic, rubber-lined steel and titanium.

Arsenopyrite plus pyrite plus carbon (double refractory)

The impact of carbon in the processing of gold concentrates was largely a function of its grade and activity. At the lower range of carbon content, either organic additives (blinding agents) were used to inhibit gold adsorption, or activated carbon was

added to the leach to preferentially adsorb gold (CIL - carbon in leach). Thus in these instances, the oxidation of the arsenopyrite was as described previously.

However, when the content of carbon starts to exceed 3 to 5%, the effectiveness of inhibition or CIL is greatly reduced as so-called "preg-robbing" of gold increased. In this instance the destruction of carbon by roasting has been the main treatment option practiced in the prior art. This is a relatively complex process, as gold extraction from the resulting calcine is affected by the roasting conditions. Further, the optimal conditions for pyrite roasting differ from those of arsenopyrite, necessitating a two stage roasting process.

The use of the IRGP prior to roasting selectively leached arsenic and sulfur to simplify subsequent roasting, which in this instance became a simpler single-stage process. Further, the removal of arsenic and sulfur reduced the duty for off-gas scrubbing from roaster operations, because As_2O_3 and SO_2 were greatly reduced. The impact was thus one of significantly reduced capital and operating costs in the roasting step.

Description of Process Flow Sheet

Referring now to Figure 2 a single refractory pyritic gold recovery process 10 is schematically depicted. A precious metal concentrate 12 for feeding to the process is prepared by mining, milling and then flotation of a sulfidic ore. The concentrate is typically a gold-containing arsenopyrite (but may have a high carbon content). The concentrate is ground in a special ball mill, typically to an ultra-fine level of less than 10 μm . The ground concentrate is then fed to a first leaching stage in the form of arsenopyrite leaching stage 14.

In the arsenopyrite leaching process 14 an acidic environment is maintained (preferably less than pH 1, as leaching of arsenopyrite is enhanced at low solution pH). The acid environment can be achieved solely by the solution recycle, or a non-contaminating acid may also be added (such as sulfuric or hydrochloric acid). The leaching solution Eh is typically maintained at greater than 0.4 volts to promote oxidation of the arsenopyritic component of the material and solubilisation of gold. The leaching temperature is maintained at around 80-95°C.

The leached material is then fed a second pyrite leaching stage 16 where an oxidant (such as oxygen, air, chlorine, hydrogen peroxide etc) is added to raise the solution oxidation potential and thereby oxidise the pyrite. To maintain arsenic in a precipitated form in the second leaching stage acid (such as sulfuric acid) and base (such as calcium carbonate) addition may be required to maintain the solution pH above about 0.2.

The process solution is typically an aqueous cupric chloride solution, having a chloride concentration of 8 moles/litre. In both the arsenic and pyrite leaching stages the cupric ion oxidises the sulfidic material and is reduced to cuprous ion (equations (2) and (8)). The cupric ion is also regenerated in the acidic oxidising environment (equations (3) and (9)). Thus, in the process, copper acts as an electron transfer agent, existing as a $\text{Cu}^{2+}/\text{Cu}^+$ couple. Other agents can perform this function, including iron, cobalt, manganese, vanadium, etc.

Where the sulfidic material has a high carbon content (eg. up to 3-5 wt%) a masking surfactant may be added to the solution at stages 14 and 16 to prevent adsorption onto carbon of any gold leached into the solution. The surfactant is typically an organic binding agent such as kerosene, a phenol ether etc. Alternatively activated carbon can be added to preferentially adsorb the gold for subsequent removal.

In arsenopyrite leaching stage 14 the present inventors have found that at a controlled pH of less than 1 but above that at which arsenic solubilises, at a controlled relatively modest Eh of about 0.7-0.8 volts (versus SHE), and at relatively low temperatures (80-95°C) the material can be leached and gold solubilised, without oxidising pyritic sulfide to sulfate.

The oxidising conditions employed in the pyrite leaching stage 16 are more severe than the arsenic leaching stage 14. In this regard an oxidant is sparged into the solution so that the oxidation potential Eh is increased to approximately 0.85 volts. In addition, the temperature of the solution in the second leaching stage may need to be raised to around 90-105°C. The pH of the solution in the second leaching stage is again controlled at less than pH 1 but above that at which arsenic solubilises.

Because acid is consumed in the second stage leaching process (ie. as Cu(II) is regenerated) it may be necessary to periodically or continuously supply acid to the solution in leaching stage 16, such as sulphuric acid, hydrochloric acid or another acid that does not interfere with the process chemistry. However, the top-up of acid depends on whether sufficient sulfuric acid is produced by the leaching of pyrite. In addition, the pH is controlled in the leaching stage 16 through the addition of calcium carbonate to prevent the solubilising of arsenic.

In leaching stage 16 sulfide is oxidised through to sulfate and iron is leached into solution as Fe(III) (equation (1)) and typically any remaining gold in the pyrite (or other precious metal) is solubilised. The inventors surprisingly observed that an oxidation potential was achievable under the influence of the Cu^{2+} and Fe^{3+} in the region of 850mV (versus SHE) in a halide solution in the presence of oxygen. This potential was sufficient for the dissolution of gold in the 8M Cl⁻ medium used, due to the formation of a gold-chloride complex.

The resultant solids slurry from pyrite leaching stage 16 is passed to a solid-liquid separation stage 18, where typically the solids are filtered from the solution using known filtering apparatus. The resulting liquid filtrate 20 is passed to an in-line precious metal recovery stage 22, whereas the filtered solids 24 are disposed of as tails. Top-up water is added at stage 18 to compensate for that lost with the tails.

The metal recovery stage 22 comprises one or more columns filled with activated carbon through which the solution is passed upwardly, such as in a fluidised bed arrangement. The solubilised gold (or other precious metal) in the solution adsorbs onto the carbon, whilst an overflow liquid stream 26 passes out of the column and is recycled to the leaching stage 14. The activated carbon bearing gold is then removed or treated periodically and passed as a gold product stream 28 to a gold recovery process (eg. by burning the carbon product or eluting the carbon column with cyanide solution).

The overflow liquid stream 26 is recycled to the leaching stage 14 via an iron precipitation stage in the form of solution conditioning stage 30. In stage 30 the soluble ferric sulfate from the pyrite oxidation stage 16 is precipitated to remove sulfur and iron from the process by the addition of limestone and calcium carbonate to form hematite and gypsum (equation (6)). Limestone addition is controlled to maintain approximately 2g/l iron in solution, to prevent the precipitation of cupric copper. The hematite/gypsum slurry is filtered and the residue washed prior to disposal to tailings. Thereafter, the solution is recycled to stage 14.

To prevent contaminant build up in the overall process, a proportion 32 of the stream 26 is recycled as a bleed circuit 34 to separate out contaminants such as Mn, Cd, Ni, Co etc (eg. through a controlled precipitation by raising of bleed solution pH).

Examples

Now that an optimal process flowpath has been described, preferred examples illustrating the process according to the present invention will be described.

Example 1

As a preliminary evaluation, the gold extraction from a first concentrate was conducted in three different steps: arseno-pyrite leach, a pyrite leach 1 and a pyrite leach 2. The following laboratory test reports describe the procedure and results of these three steps. CON1 01 covers arseno-pyrite leach and pyrite oxidation 1, and CON1 02 covers pyrite oxidation 2. The first concentrate was milled to $P_{80}=30\mu$ and subjected to an As leach followed by pyrite oxidation.

Aim

The aim was to apply the IRGP to a single refractory Au concentrate. An ore sample was provided to a metallurgical lab for milling and concentration.

Procedure

This experiment was conducted in two parts and was carried out in a 7.5 L titanium insulated tank. The first part, the As leach, used a conventional mixer. The second part, oxidation of the pyrite, used a flat blade turbine and sparger.

5 Part 1: As leach

In a 7.5 L titanium reactor fitted with a "propeller" agitator, 3.5 L of neutral brine was prepared with 200 gpl NaCl, 50 gpl CaCl_2 and $\text{pH} < 0.5$. 5 L "boost" solution was also prepared with 200 gpl NaCl, 50 gpl of CaCl_2 and 75 gpl Cu from CuCl_2 and $\text{pH} < 0.5$. As required, Eh was adjusted to between 580 and 600 mV with the addition of a few grams of copper dendrites. The boost solution was maintained at 80 °C.

After heating the leach reactor to 105 °C, the equivalent of 300 gr of dry concentrate was added to the brine. After 15 minutes, as required, concentrated HCl was added to the suspension to adjust the $\text{pH} < 0.5$ and $t = 0$ sample was taken. All additions of acid were noted (time, volume of addition, volume in leach tank).

15 Eh and pH were measured, the boost solution was added slowly at the rate of 2.5 l/hr, monitoring the Eh in order not to exceed 530 mV. Samples of solution were taken every 30 minutes for As, Fe, Cu analysis. Eh & pH were monitored every 30 minutes.

When an Eh of 530 mV was reached and was stable, the As leach was considered as complete. The slurry was filtered. The cake was washed twice with hot brine (50 gpl NaCl and $\text{pH} < 1.0$) followed with hot water washes until the filtrate was clear. The cake was dried in an oven overnight. The cake was analysed for $\text{S}_{(T)}$, $\text{S}_{(E)}$, As, Fe, Au and C.

Part 2: Pyrite Oxidation

25 A 7.5 L reactor was equipped with a flat blade turbine agitator and a titanium sparger tube. 10 litres of brine solution was prepared in the leach tank with 200 gpl NaCl, 50 gpl of CaCl_2 and 75 gpl Cu from CuCl_2 and $\text{pH} < 0.5$ with addition of concentrated HCl 8.8 molar. The solution was heated to 105 °C, $t=0$ sample was taken and the dry cake produced in part 1 As leach was introduced into the tank. After 15 minutes, a sample of solution was taken for Eh and pH measurement. Technical HCl was added as required to bring the $\text{pH} < 0.5$.

30 Oxygen was introduced at the rate of 2 l/min; Eh and pH were monitored every 30 minutes, and samples were taken every hour for Fe, As, Cu analysis. When the Eh was stable above 600 mV for 3 hours and the Fe in solution did not change, the leach was considered as complete. The slurry was filtered. The cake was washed twice with hot brine (50 gpl NaCl and $\text{pH} < 1.0$) followed with hot water washes until the filtrate

was clear. The cake was dried in an oven overnight. The cake was analysed for $S_{(T)}$, $S_{(E)}$, As, Fe, Au and C.

Example 2

5 An additional pyrite oxidation of the concentrate residue from Example 1 was conducted.

Aim

10 The analysis of the data and the residue from Example 1 showed that the pyrite oxidation was not completed when the experiment was terminated. This procedure with an improved brine formula attempted to increase Au extraction using oxygen to oxidise the pyrite.

Outcome

The second pyrite oxidation improved metals extraction as shown in the following table (based on independent analysis):

Elements	Oxidation 1	Oxidation 2
As	79.6 %	92.4 %
Fe	72.2 %	97.1 %
Au	68.7 %	93.3 %

15 Procedure

A 7.5 L reactor was equipped with a flat blade turbine agitator and a titanium sparger. 5 litres of brine solution was prepared in the leach tank with 100 gpl NaCl, 250 gpl of $CaCl_2$ and 100 gpl Cu from $CuCl_2$ and $pH < 0.5$ with the addition of concentrated HCl. The solution was heated to $105^\circ C$, $t=0$ sample is taken and the dry cake produced in Example 1. As leach / Pyrite oxidation was introduced into the tank. After 15 minutes, a sample of solution was taken for Eh and pH measurement. Concentrated HCl was added if required to bring the $pH < 0.5$.

20 Oxygen was introduced at the rate of 2 l/min, Eh and pH were monitored every 30 minutes and samples were taken every hour for Fe, As, Cu analysis. When the Eh was stable above 600 mV for 3 hours and the Fe in solution did not change, the oxygen flow was interrupted and the Eh monitored. When the Eh stayed above 600 mV, the pyrite oxidation was considered completed.

25 The slurry was filtered. The cake was washed twice with hot brine (50 gpl NaCl and $pH < 1.0$) followed with hot water washes until the filtrate was clear. The cake was dried in the oven overnight. The cake was analysed for $S_{(T)}$, $S_{(E)}$, As, Fe, Au and C.

Results

30 The following results were obtained from the experiments of Examples 1 & 2.

	Duration	T	Eh	pH	Fe Cum	As Cum	Fe	As
	hr	°C	mV		g	g	g/l	g/l
Arsenopyrite Leach	0.0	105	#N/A					
	0.3	105	#N/A	0.4	2.5	0.0	2.5	0.00
	0.7	105	505	< 0.5	2.8	0.00	2.8	0.00
	1.2	105	512	< 0.5	3.3	0.00	3.3	0.00
	1.6	105	518	< 0.5	3.6	0.00	3.6	0.00
	2.1	105	527	< 0.5	4.0	4.16	4.0	0.42
	2.4	105	525	< 0.5	4.3	4.11	4.3	0.41
	2.9	105	528	< 0.5	4.4	4.12	4.4	0.41
	3.4	105	531	0.2	5.0	4.20	5.0	0.42
Pyrite Oxidation 1	0.25	3.7	105	580	0.4	6.0	4.20	1.0
	1.25	4.7		602	0.5	7.8	4.20	2.9
	2.25	5.7	105	606	0.6	9.6	4.20	4.6
	3.25	6.7	105	597	0.5	12.0	9.20	7.0
	4.25	7.7	105	602	0.5	15.0	9.20	10.0
	5.25	8.7		607	0.4	17.0	9.70	12.0
	6.25	9.7	105	606	0.5	18.5	9.20	13.5
	6.75	10.2	105	620	0.5	18.5	9.20	13.5
	9.75	13.2	105	609	0.7	21.2	11.20	16.3
	10.75	14.2	100	616	0.7	22.7	11.70	17.8
	11.75	15.2	100	616	0.6	24.7	12.20	19.8
	12.75	16.2	105	612	0.6	26.2	13.20	21.3
	13.75	17.2	105	623	0.6	26.7	13.20	21.8
	15.75	19.2	105	625	0.6	28.5	13.20	23.5
P	0.75	19.9	105	661	0.2	29.6	13.20	0.5
	1.75	20.9	105	661	0.2	31.7	13.20	1.5
	2.75	21.9	105	661	0.3	32.8	13.20	2.0
	3.75	22.9	105	674	0.3	33.9	13.20	2.5
	4.75	23.9	105	664	0.5	36.1	13.20	3.5
	5.75	24.9	104	665	0.4	37.2	13.20	4.0

7.25	26.4	102	670	0.4	38.3	13.20	4.5	0.00
8.25	27.4	104	675	0.4	39.4	13.20	5.0	0.00
9.25	28.4	103	673	0.3	39.4	13.20	5.0	0.00
10.25	29.4	103	671	0.4	40.5	13.20	5.5	0.00
12.25	31.4	105	669	0.7	41.6	13.20	6.0	0.00
13.25	32.4	105	673	0.7	41.6	13.20	6.0	0.00
13.75	32.9	105	664	0.8	41.6	13.20	6.0	0.00

The results of gold extraction are displayed in Figure 3.

Example 3

5 In this experiment, the gold extraction from a second concentrate was achieved in three consecutive steps: step 1 arseno-pyrite and pyrite leach, step 2 pyrite leach with oxygen, and step 3 pyrite leach with chlorine.

Aim

10 Following a scouting experiment on As leach, this procedure aimed to treat the second concentrate in an "all-in-one" process with a CaCl_2 250 g/l and Cu 100 g/l brine. The solid loading was set at 200 g/l.

Outcome

The chlorine oxidation improved Au extraction as shown in the following table (based on external analysis):

	Au Extraction (cumulative)
Air + Oxygen	59 %
Chlorine	87 %
Overall	95 %

15

Procedure

The procedure was carried out in a 7.5 L reactor equipped with a turbine agitator. 5 l of brine was prepared with the following formula: NaCl 100 g/l, CaCl_2 250 g/l and Cu 100 g/l and pH was adjusted to < 0.5 with the addition of concentrated HCl .

20

Part 1: Arseno-pyrite leach

With the agitator rpm at 90%, the brine was heated to 90 °C. A sample of solution was taken for future reference. 1,000 g of equivalent dry "as-received"

concentrate (P80 around 37μ) was added to the brine. A sample $t=0$ was taken, and after 15 minutes as Eh & pH were recorded.

Air was introduced into the reactor at the rate of 2 l/min. Eh, pH were monitored every 30 minutes as a solution sample was taken for As, Fe analysis. When Eh and Fe in solution were stable, the airflow was interrupted. If the Eh dropped by more than 20 mV, the air introduction was resumed for 2 hours. When the Eh did not drop by more than 20 mV, a solid sample of approximately 100 g was taken and a switch was made from air to oxygen.

Part 2: Pyrite oxidation

The temperature was increased to 105 °C. Sample and measurement frequency was changed to a 1-hour interval. Oxygen was introduced underneath the turbine agitator at the rate of 2 l/min. When the Eh and Fe in solution were stable, the oxygen was interrupted. If the Eh dropped by more than 20 mV, the oxygen introduction was resumed for 2 hours. When the Eh did not drop by more than 20 mV, the procedure was considered complete.

The suspension was filtered, the cake washed twice with acidic brine followed by hot water until a clear filtrate was obtained. The washed cake was dried and weighed. The residue was analysed for As, Fe, Cu, elemental S, total S and Au. The last solution sample was also analysed for Au.

Part 3: Pyrite Chlorination

In order to improve Au extraction, the experiment was extended with a pyrite chlorination using hypochlorite as the source of chlorine. The residue from the pyrite oxidation with oxygen was introduced in 4 litres of brine with the same composition as above. The temperature was raised above 100°C and 50 g of hypochlorite was added every period of 30 minutes. Fe concentration was monitored. When Fe concentration did not increase after hypochlorite addition and the Eh was stable, the experiment was considered complete.

The suspension was filtered, the cake washed twice with acidic brine followed by hot water until a clear filtrate was obtained. The washed cake was dried and weighed. The residue was analysed for As, Fe, Cu, elemental S, total S and Au. The last solution sample was also analysed for Au.

Results

	Duration (hr)	Eh mV	pH	Fe g/l	Tot Fe g
Air	1.1	464	0.41	11.2	56.0 g
	2.0	479	0.36	18.9	94.5 g

Oxygen	3.0	470	0.23	20.3	101.5 g
	4.0	477	0.49	19.8	99.0 g
	5.0	473	0.49	18.9	94.5 g
	6.5	473	0.41	20.2	101.0 g
	7.5	472	0.53	22.4	112.0 g
	8.5	474	0.05	22.0	105.6 g
	9.5	473	0.53	20.0	100.0 g
	10.5	482	0.61	20.4	112.2 g
	12.0	494	0.38	21.4	117.7 g
	13.0	485	0.71	21.0	115.5 g
	14.0	492	0.65	20.3	111.7 g
	15.0	491	0.71	22.0	121.0 g
	17.0	500	0.22	23.0	124.2 g
	18.0	501	0.52	20.6	109.2 g
	21.0	517	0.15	23.3	121.2 g
Hypochlorite	22.5	522	0.59	21.3	106.5 g
	23.5	537	0.64	18.7	93.5 g
	24.5	552	0.64	16.3	81.5 g
	26.0	571	0.86	14.3	71.5 g
	27.0	613	0.69	14.8	74.0 g
	28.0	645	0.73	15.8	79.0 g
	28.5	630	0.77	16.2	164.8 g
	31.0	661	0.35	17.7	172.7 g
	32.5	660	0.40	17.8	173.2 g
	33.5	691	0.10	18.0	174.3 g
	34.5	667	<0.1	21.7	193.9 g
	35.5	664	<0.1	21.3	191.8 g
	37.0	667	<0.1	21.7	193.9 g
	39.0	684	#N/A	23.5	203.4 g
	40.0	#N/A	#N/A	21.3	191.8 g

The results of gold extraction are displayed in Figure 4.

Whilst the invention has been described with reference to a number of preferred embodiments, it will be appreciated that it can be embodied in many other forms.

CLAIMS

1. A process for recovering a precious metal from a sulfidic material comprising the steps of:
 - preparing an acidic aqueous halide solution having an oxidation potential sufficient to oxidise the sulfidic material and render the precious metal soluble in the solution;
 - adding the material to the acidic aqueous halide solution so that the sulfidic material is oxidised and the precious metal is solubilised; and
 - separating the precious metal from the oxidised sulfidic material.
2. A process for recovering a precious metal from a sulfidic material contaminated with arsenic comprising the steps of:
 - preparing an acidic aqueous halide solution having an oxidation potential sufficient to oxidise the sulfidic material and render the precious metal soluble in the solution, and having a pH at which the arsenic is precipitated;
 - adding the material to the acidic aqueous halide solution so that the sulfidic material is oxidised, the precious metal is solubilised and the arsenic is precipitated; and separating the precious metal from the oxidised sulfidic material and precipitated arsenic.
3. A process as claimed in claim 1 or 2 wherein the solution bearing the precious metal is separated from the oxidised sulfidic material and precipitated arsenic (when present) in a solid-liquid separation stage, and the precious metal is then recovered from the solution in a metal recovery stage.
4. A process as claimed in claim 3 wherein in the metal recovery stage the precious metal is adsorbed onto activated carbon in one or more carbon-containing columns.
5. A process as claimed in claim 4 wherein after precious metal adsorption onto activated carbon the carbon is eluted with a cyanide solution and the eluate is passed to an electrolysis stage for the recovery of the precious metal.
6. A process as claimed in any one of claims 3 to 5 wherein the metal recovery stage is provided in-line, after the solid-liquid separation stage, and prior to solution recycle to sulfidic material oxidation.
7. A process as claimed in any one of the preceding claims wherein the precious metal to be recovered is gold, silver, platinum or another platinum group metal.

8. A process as claimed in any one of the preceding claims wherein the aqueous halide solution is a soluble metal halide solution having a halide concentration of approximately 8 moles per litre.

9. A process as claimed in claim 8 wherein the halide is chloride or a mixture of halides comprising chloride and bromide.

10. A process as claimed in claim 8 or 9 wherein the metal in the dissolved metal halide solution is copper and/or iron and functions as a multi-valent species.

11. A process as claimed in any one of the preceding claims wherein the sulfidic material oxidation step comprises one or more leaching stages such that:

(i) for an un-contaminated single-refractory pyritic material the sulfidic material oxidation step comprises a single leaching stage in which the pyritic material is oxidised and the precious metal simultaneously solubilised; or

(ii) for a contaminated single or double refractory pyritic material, the sulfidic material oxidation step comprises a two stage leaching process wherein the solution from the first leaching stage is fed to the second leaching stage.

12. A process as claimed in claim 11 wherein for (ii) the pyritic material is an arsenopyrite, and in a first of the leaching stages the oxidation potential is controlled to leach arsenic into solution and the solution pH is controlled such that, once leached, the arsenic precipitates as ferric arsenate, and in a second of the leaching stages the pyrite component is leached and the solution pH is controlled to maintain arsenic as a ferric arsenate precipitate so that the arsenic passes out of the process with the oxidised sulfidic material.

13. A process as claimed in claim 12 wherein in the first leaching stage the material is contacted with solution at an Eh of around 0.7-0.8 volts sufficient to leach the contaminant and solubilise the precious metal, at a solution pH is less than 1 but greater than about 0.5 so as to precipitate the arsenic immediately after it is leached, and at a solution temperature of about 80-105°C.

14. A process as claimed in claim 12 or 13 wherein in the second leaching stage the material is contacted with a solution having an Eh of around 0.8 - 0.9 volts sufficient to leach pyrite, the solution pH is less than 1 but greater than about 0.2 so as to precipitate

the arsenic immediately after it is leached, and at a solution temperature about 90°C to 105°C

5 15. A process as claimed in any one of the preceding claims wherein after precious metal recovery a solution conditioning stage is employed to precipitate ferric sulfate and thus control the level of this species in the process.

10 16. A process as claimed in claim 15 wherein in the solution conditioning stage limestone and calcium carbonate are added to the solution to form a hematite/gypsum precipitate which is then filtered and disposed of with the solids residue from the leaching stage(s).

15 17. A process as claimed in any one of the preceding claims wherein, when a high level of carbon is present in the sulfidic material, a surfactant is added to the solution during the sulfidic material oxidation step to prevent precious metal from adsorbing onto carbon in the material, or activated carbon is added to the solution during the sulfidic material oxidation step to preferentially adsorb precious metal onto the activated carbon.

20 18. A process as claimed in claim 17 wherein the surfactant is one or more organic solvents including kerosene or a phenol ether.

19. Any metal produced by the process of any one of the preceding claims.

25

Dated this 20th day of June 2003

Intec Ltd

By its Patent Attorneys

GRIFFITH HACK

O X I D E

(pH > 9)

S U L F I D E

(pH < 2)

FIG. 1

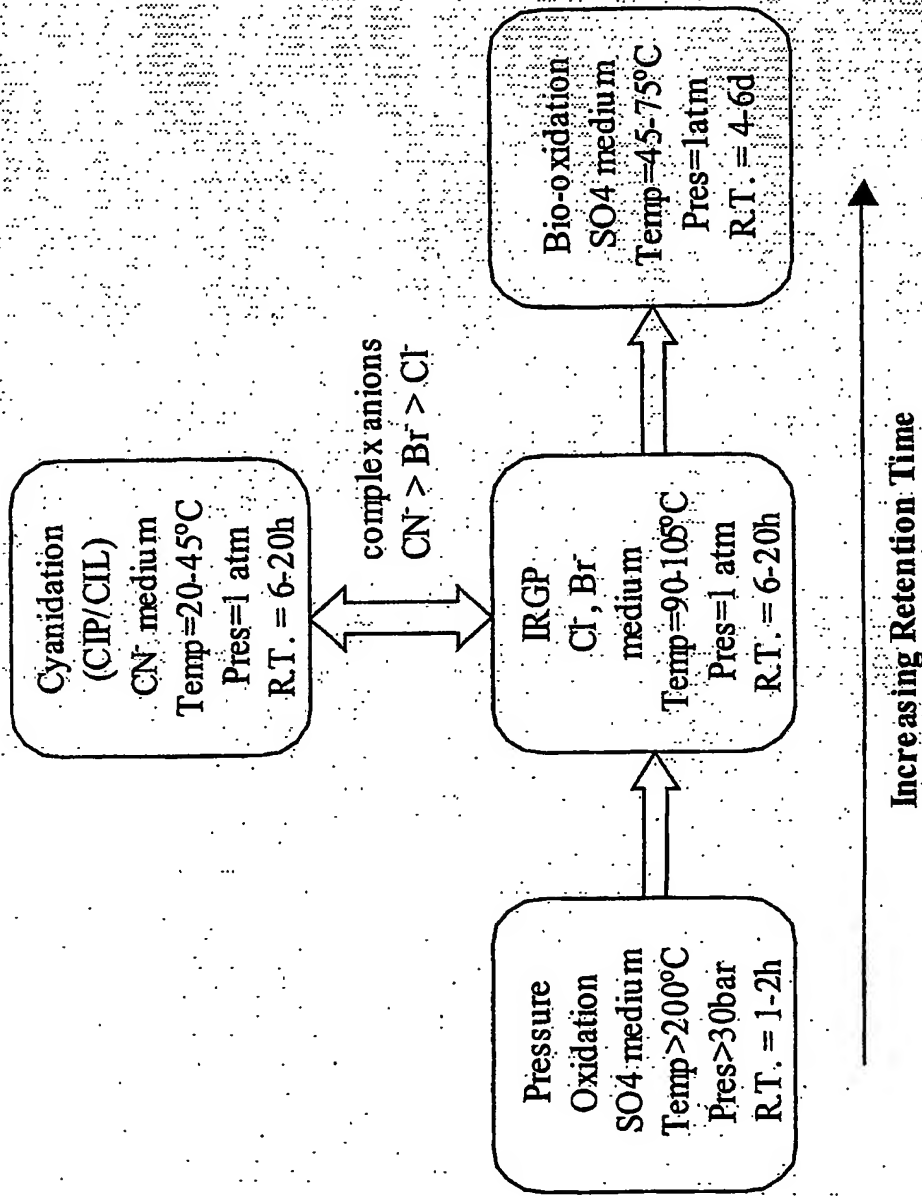


FIG. 2

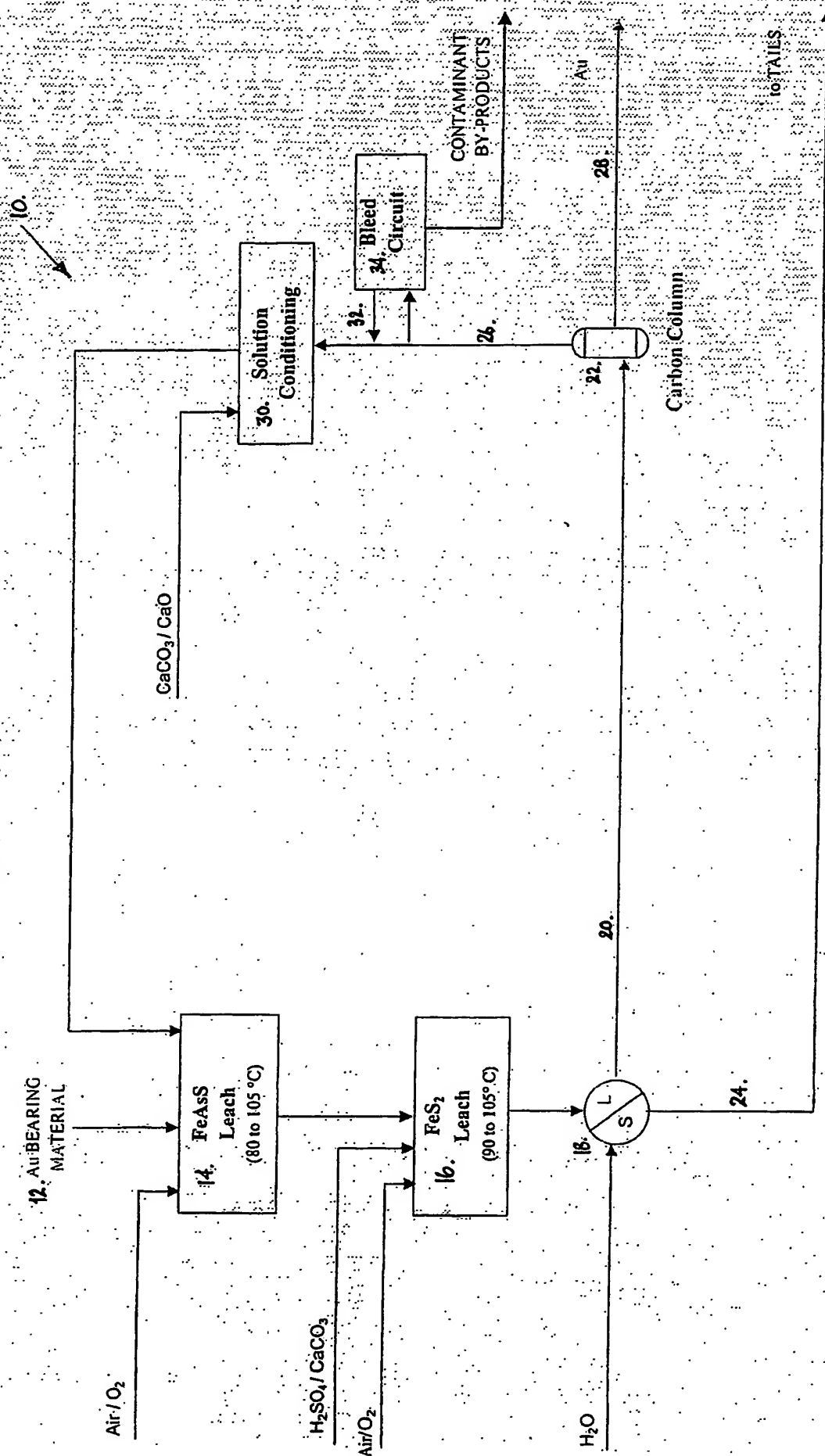


FIG. 3 Au Extraction

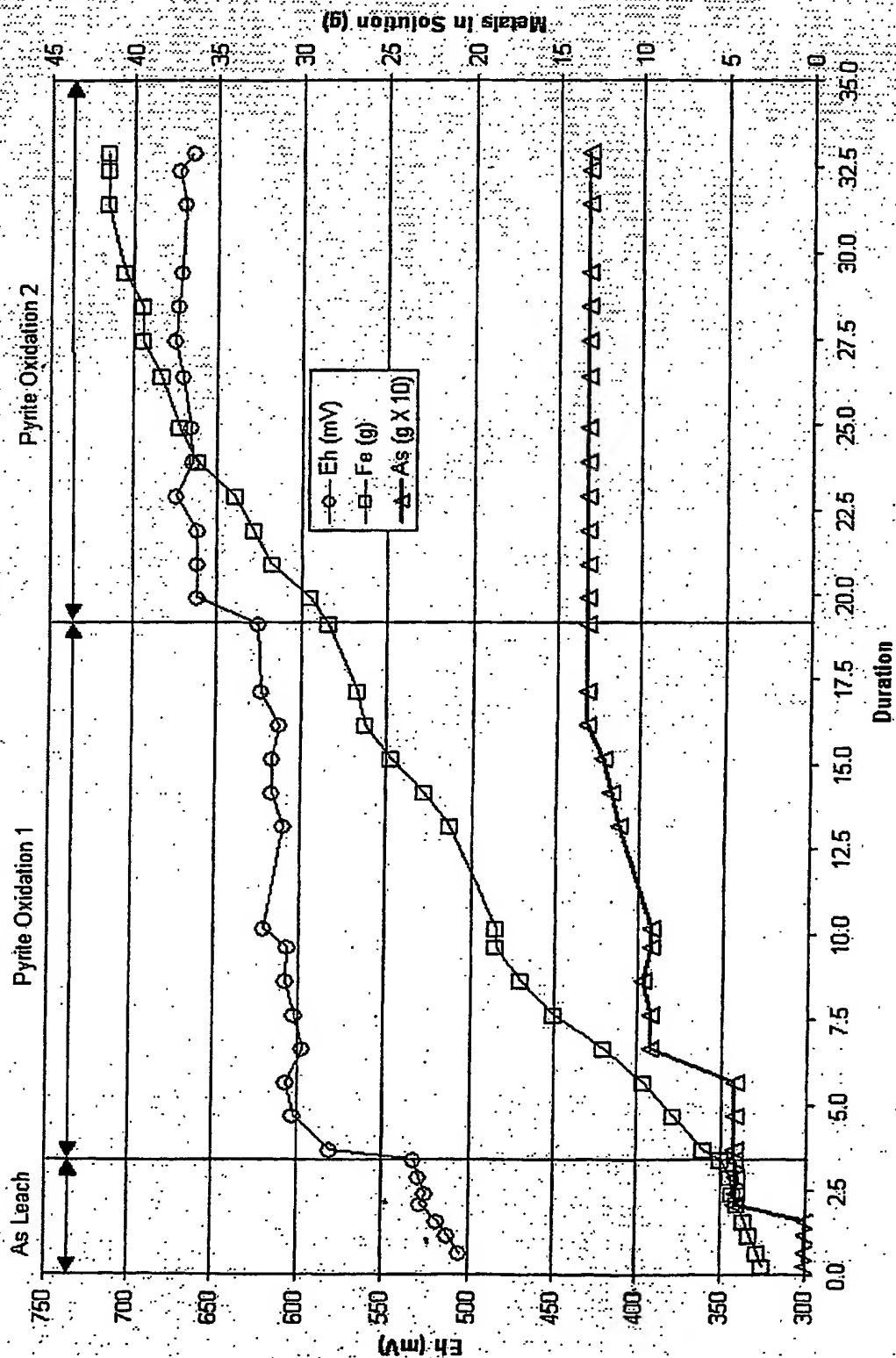
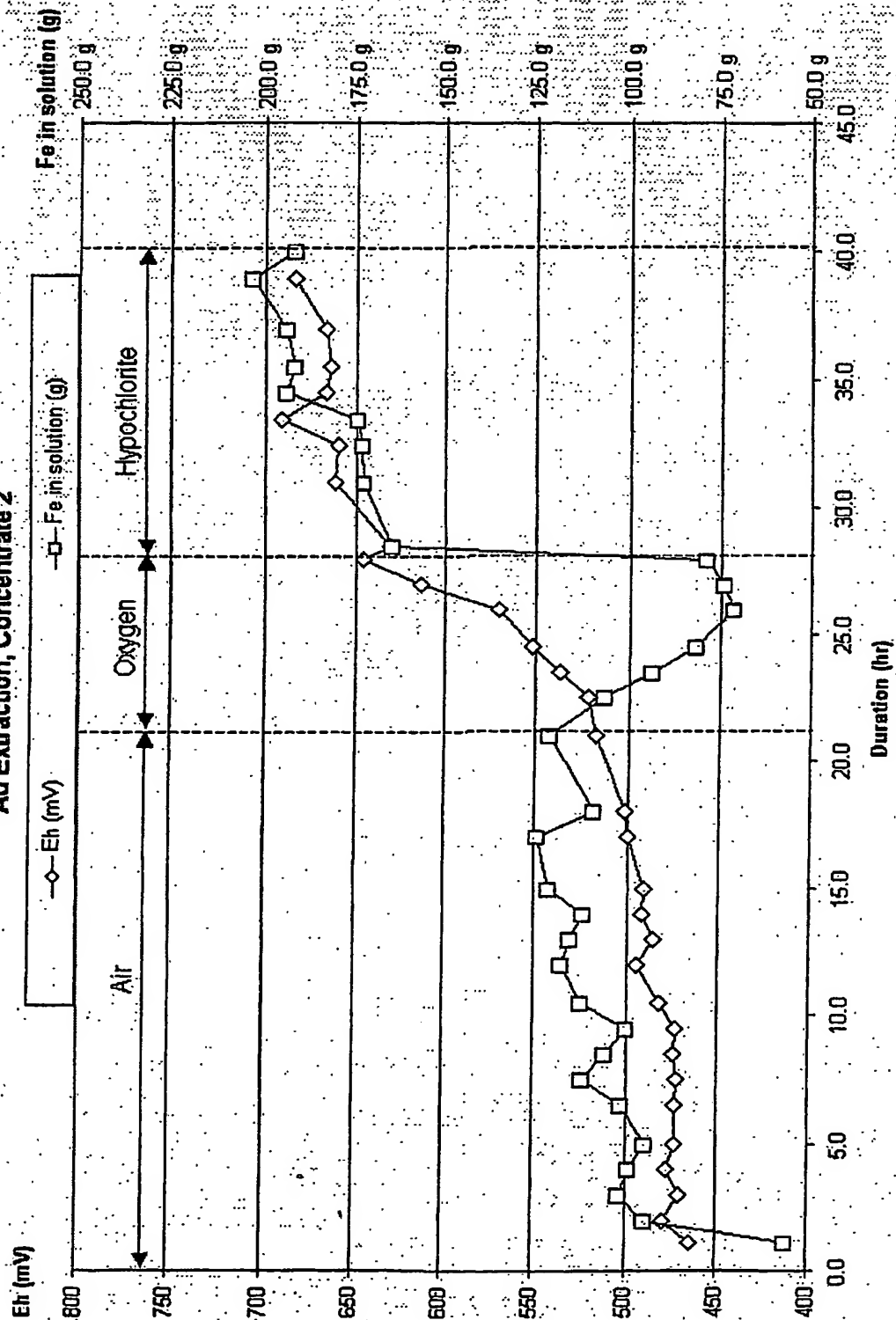


FIG. 4

Au Extraction, Concentrate 2



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